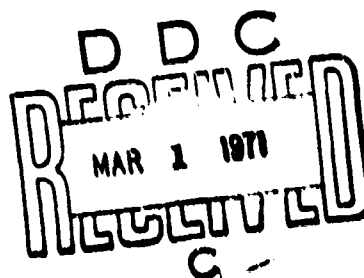AD718886

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THE VACUUM THERMAL STABILITY TEST FOR EXPLOSIVES

By
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NAVAL ORDNANCE LABORATORY, WHITE OAK, SILVER SPRING, MARYLAND

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THE VACUUM THERMAL STABILITY TEST
FOR EXPLOSIVES

Prepared by:

Herbert T. Simmons, Sr.

ABSTRACT: This report covers a complete description of the methods and procedures used in the vacuum thermal stability test. It describes the construction and assembly of the test chamber, installation of the two heating elements, electronic temperature controller, thermocouple monitoring system, glass sample apparatus, sample tube calibration, and a compatibility test evaluation.

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THE VACUUM THERMAL STABILITY TEST FOR EXPLOSIVES

This report describes the vacuum thermal stability test used to
evaluate high explosives. Most of this work has been supported
by ORDTASK ORD-332-001/UF17-354-301, Explosives Characterization.
This evaluation method has been found to be extremely useful for
determining the suitability of explosives for use when exposed to
high temperatures. It also has value in determining the compati-
bility of explosives with other materials with which they may come
in contact during a high temperature stability but with a knowledge
of the ingredients and their possible reactions, it can lead to a
method of specification and selection of explosives for specific
applications.

                              GEORGE G. BALL
                              Captain, USN
                              Commander

                              ALBERT LIGHTBODY
                              By direction

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INTRODUCTION

Supersonic aircraft and missile development and space exploration required the synthesis of new heat resistant explosives. These new explosives, most of which were discovered at the Naval Ordnance Laboratory, were found to be stable at high temperatures. In our thermal stability evaluation program new equipment had to be designed to test efficiently these explosives in the range of 200 - 350°C, as the standard test method was not suitable.

A new test chamber was built (1) (2) which has been modified several times; the old style stability glass tube and manometer which were joined together with a standard taper joint was discarded in favor of a one-piece sealed glass tube and manometer (Fig. 1).

Tests are conducted at elevated temperatures to obtain data rapidly which indicate the explosives resistance to storage environment. The vacuum stability method measures the amount of gas evolved at a fixed temperature. A relatively small weight of solid decomposing will usually yield a substantial volume of gas. In addition to pointing out inherently unstable compounds, the method also distinguishes between pure and impure samples of the same compounds. Decomposition which occurs without gas evolution will not be revealed by this test.

A number of successful studies have been carried out using the equipment and methods described herein, including the development of new explosives such as DATB and HNS; studies concerning the relationship between molecular structure and thermal stability of a related series of compounds (3); and the compatibility evaluation of a wide variety of materials with explosives, including inorganic azides with organic explosives (4). Another example is the discovery that two of the heat resistant explosives decomposed in the vapor phase as well as in the solid.

In the development of explosive mixtures for specific purposes, the explosive system must yield the desired results and retain its properties through various terms and conditions of storage. Compatibility testing using the vacuum stability procedure and test equipment has been used for years to screen materials for specific explosive systems. Tests are run under extreme conditions of temperature. Mixtures that pass the test (no chemical interaction or gaseous decomposition) are compatible and can be expected to have a long storage life. When there is evidence of an interaction, as shown by decreased thermal stability of the mixture, the system is incompatible.
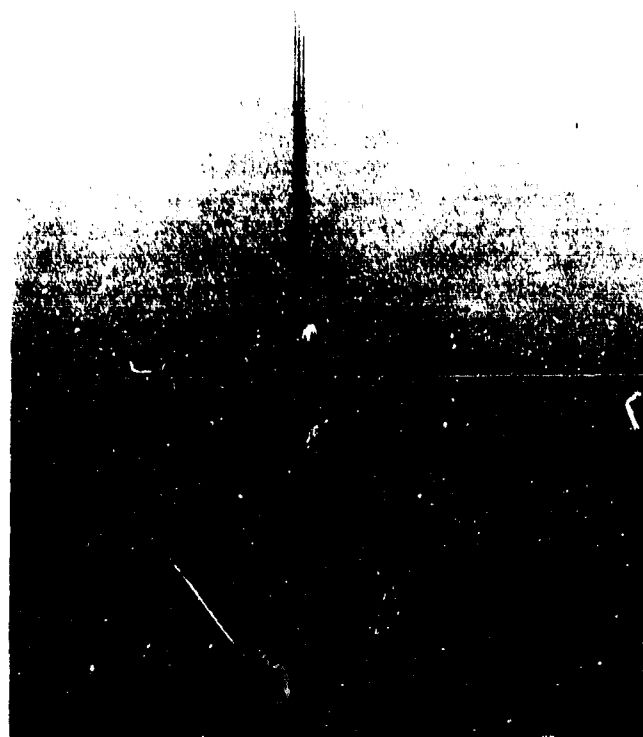
FIG. 1

DESCRIPTION AND ASSEMBLY OF TEST CHAMBER

One of the major objectives in designing the new test chamber was to keep the heat loss at an absolute minimum. A "lazy-susan" type revolving top is used in which only the cover plug is removed to insert or remove a sample tube. This exposes only one hole at a time and the top can be rotated so that the plug is not directly over any sample hole.

1. The test chamber (Fig. 2) essentially consists of a cylindrical aluminum block 8" O.D. and 12" long designated sample holder (Dwg. No. SE/SK 4008). This sample holder has 12 sample holes 1.125" diameter drilled to a depth of 8 inches in the top on a 6.0" circle diameter. Three holes are drilled 120° apart on a 7.0" diameter circle between sample holes. These holes are tapped with a 10-24 NC thread and are used to mount the top aluminum plate to the sample holder. A 1.125" diameter hole is drilled into the side of the sample holder just below one of the sample holes and deep enough so that all of the spiral sensing part of the thermoregulator is inside the sample holder. A 0.5" diameter hole for a thermometer is drilled on a 30° angle directly into the side of a sample hole. This hole should be drilled so that the bulb of a thermometer will be at the same depth as the sample. The thermometer hole is generally not used on test chambers designed for testing heat resistant explosives. Thermoregulator and thermometer holes are bored large enough for Mullite protection (MV-30) tubes that are used as sleeves. Two holes are drilled on the side of the block one on each end for flush mounting of ceramic lead-in bushings used for anchoring the nichrome heating element and lead wires. Holes are centered between sample holes and are drilled with center of hole being 0.5" from top and bottom of sample holder respectively. These holes are drilled with a 0.875" cutter with a 90° point. Depth of 0.875" diameter cut being 0.437" deep. Holes are tapped for a 6-32 machine screw thread for mounting ceramic lead-in bushings.

Efficient heat control is obtained by providing an air space between the two heaters. This is accomplished by separating the sample holder and the top aluminum plate by six Lamicoid washers made from 1/16" stock material. These washers are countersunk for 4-40 machine screws and are mounted on top of the sample holder 60° apart on a 5.0" diameter bolt circle.

In section c-c of sample holder Dwg. No. SE/SK 4008 provision is shown for the termination of the ground wire.

2. Main or bottom heating element.
Several layers of thin asbestos paper or a piece of asbestos cloth is wrapped tightly around the sides of the sample holder. The heating element consists of 60 feet of AWG No. 16 nichrome wire spiral wrapped equally spaced (approx. 0.375" pitch) around the asbestos insulation. A loop can be silver soldered on starting end of nichrome wire. This can be anchored to one of the ceramic lead-in bushings. The sample holder is placed in a large lathe and by means of a guide and automatic feed the wire is wound tightly around the sample holder and secured on the other ceramic lead-in bushing. Two asbestos covered AWG No. 12 lead wires with silver soldered loops are hooked up to the ceramic lead-in bushings in contact with the heating element wire.
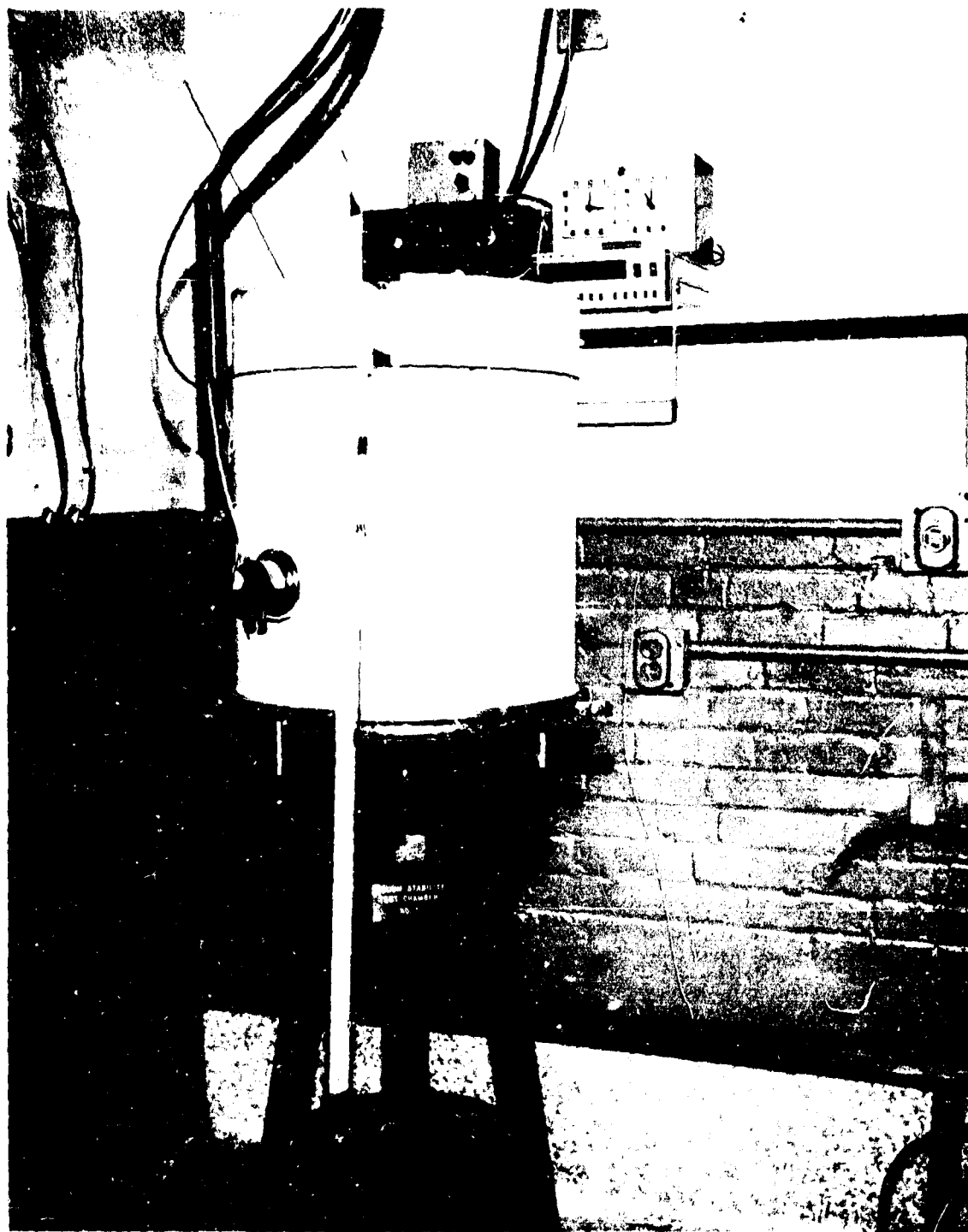
FIG. 2

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3. Assembly of bottom part of Test Chamber.

The outer container made in two parts is fabricated from a material formerly called Lamicoid (MIL-P-997D). This is a plastic sheet glass cloth laminated with a silicone resin. The bottom cylindrical part has dimensions of 17.0" I.D., 16.0" deep, and a wall thickness of 0.250". Heat shield or insulating material is an asbestos type (Marnite 36 type A) which will stand a continuous heat to 482°C. This Marnite can be cut from 1" stock or it can be purchased already cut to desired dimensions. It machines neatly on a lathe.

Two solid Marnite discs 17.0" were placed in bottom of outer container, and a smaller 14.0" O.D. solid Marnite disc centered on top of the above disc. Sample holder was placed on top of this smaller disc. The 12 inner Marnite rings 14.0" O.D., and 8.0" I.D., were slipped down over sample holder. It was necessary to cut the holes during assembly for Mullite tubes in the inner and outer Marnite rings, reflective shield, and the outer container. Grooves will also have to be cut for the asbestos covered lead wires.

A chrome plated reflective 26 gage, (.010" thick) steel shield with dimension of approximately 12.875" x 43.0" (Dwg. No. SE/SK 4007) is placed around the Marnite inner rings and the smaller solid disc below sample holder.

The 14 outer Marnite 17.0" O.D. rings were slipped down over the reflective shield. Before the 14th ring is installed the top (1" thick by 14.0" O.D.) aluminum plate is bolted to the sample holder. This aluminum plate (Dwg. No. SE/SK 2267) has 12 holes that match those in the sample holder; circular slots 0.375" wide and 0.375" deep with a tangent radius are equally spaced for the horizontal part of the glass manometer, a center recess 4.52" diameter and 0.9" deep for a 4.50" diameter thrust ball bearing, and a groove machined on the under side for a tubular type of heating element. A special 40" long tubular heating element is embedded in the 0.328" wide machined groove on a 12.5" diameter circle with a 1.250" lead in and lead out radius. Leads for the tubular heating element are silver soldered onto both ends of the heating element. Leads are made from same asbestos covered cooper wire as used for the main heating element. The 14th outer Marnite ring is slotted to match those in the top aluminum plate. Slots will also have to be filed or ground in the outer container to match those in the top aluminum plate and the 14th outer Marnite ring. Care is taken to see that all Marnite insulation is assembled with no "slop".

Leads for both heating elements are terminated in a covered barrier-type terminal block or a junction box made from 0.250" flat Lamicoid stock containing two male plugs for external hook-up. The junction box is mounted on the outside of outer container between manometer slots. Bottom section of test chamber is now assembled.

4. Top part of Test Chamber.

The test chamber cover assembly (Dwg No. SE/SK 4002) is constructed as follows: The outer Lamicoid container with the same dimensions as bottom part except four inches deep is placed inverted on a flat surface and three solid Marnite 17.0" O.D. discs are placed in it. The 1.0" thick by 14.0" O.D. aluminum bottom plate is placed upon the solid Marnite disc, and an outer Marnite ring 17.0" O.D. by 14.0" I.D. is slipped over the aluminum plate. The aluminum plate designated bottom aluminum plate (Dwg. No. SE/SK 2267) has three holes drilled

on a 8.0" diameter bolt circle 120° apart and 0.750" deep on the top side. These
holes are tapped for 1/4-20 NC machine screws. A 1.0 wide slot is cut from the
outside edge through the plate 4.0" toward the center and finished off with a
tangent radius. This cut is for the cover plug which is used for removing and
inserting samples. The underside of this plate has a center recess 4.520" dia-
meter and 0.205" deep for the top part of the ball bearing. A 1.125" wide slot
or recess is machined on the under side 0.312" deep on a 6.0" diameter circle.
This recess which when assembled is directly over the sample holes allows for
protruding glass tips on sample tubes when sealed a little too high. All parts
in the test chamber top are tied together so that it can be lifted as a unit,
and when assembled and revolving on the ball bearing all surfaces on the bottom of
the top part of test chamber should be in the same plane. Before mounting the
solid Marnite disc to the bottom aluminum plate three bolts are placed through
it with the bolt heads counterbored in the Marnite and next to the aluminum plate.
This Marnite disc is then secured to the aluminum plate by means of three other
bolts (1/4-20 NC) that are screwed into the tapped holes in the aluminum bottom
plate. The other two solid Marnite discs which have three holes previously drilled
are slipped down over the three bolts. The top solid Marnite disc has counter-
bored holes for nuts that are used on the three bolts. This ties the bottom
aluminum plate and the three solid Marnite discs together. This sub-assembly
is now placed upon a band saw and using the bottom aluminum plate which already
has a machine cut for the cover plug hole can be used as a template. A 1" wide
cut is made through the Marnite, finishing off the cut with a tangent radius just
like the aluminum bottom plate. The outer top Lamicoid container was cut on a
band saw for the cover plug hole. This cut was made approximately 2" wide with
an equally larger tangent radius than the cover plug hole. The cut out piece was
used as the top part of the cover plug. All parts were placed back in the inverted
outer container and three holes are drilled through the outer container, solid
Marnite discs, and Marnite outer ring. One hole being 180° from cover plug hole
and through middle of outer Marnite ring. The outer Marnite ring was counterbored
for plastic nuts. Lamicoid 0.375" rods were used for bolts, and plastic nuts can
be made from 0.250" Lamicoid flat stock. The nuts were tapped for a 3/8-16 thread,
but the bolts will have to have threads ground on them. Plastic nuts were placed
on one end of the Lamicoid bolts and inserted up through the outer Marnite ring,
Marnite solid discs and outer container. Lamicoid nuts were put on the bolts on
the outside of the outer container and drawn up snug.

The cover plug assembly (Dwg. No. SE/SK 4013) was made from 1.0" Marnite
stock. This 1 x 4 x 6" overall piece of Marnite has a tangent radius on one end
to fit snugly in the hole cut through the top part of the test chamber. This
Marnite plug along with the piece cut out of the top outer container and a handle
made from a non-metallic material are all bolted together to make up the cover
plug. The cover plug when in place fits the hole rather snugly, and the piece
cut from the Lamicoid outer container overlaps the hole thereby holding down heat
losses by covering up air spaces between cover plug and other parts.

The de-greased ball bearing was thoroughly coated with graphite and was
placed in the recess on the top aluminum plate. The top part of the test chamber
was then lowered down over the ball bearing and the test chamber is now completely
assembled.

Before the test chamber can be put into operation the Mullite sleeves
were inserted into their respective holes and the thermoregulator was installed
in the test chamber. A centering device (Dwg. No. RK/RK 7145) was mounted on
the thermoregulator shaft before it was installed, and the thermoregulator was
mounted to the test chamber by means of a circular piece of 1" thick Marnite
same diameter as thermoregulator flange. This circular piece of Marnite was
shaped to fit the curvature of the outer container and was bolted to it with
other bolts securing the thermoregulator to the Marnite.

TEMPERATURE CONTROL

The vacuum stability test chambers can be controlled from $35^{\circ}C$ to $350^{\circ}C$,
with a temperature stability of about $\pm 0.2^{\circ}C$. Test temperature heat is supplied
by two elements. The lower unit consists of 60 feet of No. 16 nichrome wire (15
ohms), and the upper unit is a tubular (50 ohm) element.

Continuous a-c power is used to obtain about 95 percent of the desired tem-
perature and temperature control is obtained by shorting out a series resistor
to each of the heating elements. A moderately simplified temperature control
circuit is shown in Figure 3. The upper and lower elements are labeled 50 ohm
and 15 ohm, respectively. The bi-metal sensor via the electronic controlled
relay controls the shorting out of the two resistors. The series resistor sizes
are such that from a no shorting to a continuous shorting condition a temperature
rise of about 12 percent is possible. In practice the continuous a-c power is
adjusted by two variacs for constant temperature, and the continuous a-c power
level is such -- usually about 95 percent of required temperature -- that the
bi-metal sensor is demanding a temperature increase 50 percent or less of the
time. The 50 percent or less shorting time insures adequate control against
a-c line and ambient temperature changes. The tubular heating element is re-
quired in order to maintain constant temperature throughout the hot zone of the
sample unit. The tubular element cuts down on the gradient heat loss up the
sample tube, and it heats the capillary part of the sample tube that is in the
test chamber. The tubular element was also installed to aid in controlling
sample vaporization, sublimation, and distillation encountered with some samples.
The horizontal part of the sample test unit can now be heated slightly higher
than the test temperature in an effort to keep all the sample in the hot zone
during the test.

The temperature control has a built-in safety device, such that in case of
equipment failure the test chamber cannot overheat and cause explosive samples
to explode. This is accomplished by selecting a voltage that would heat the
test chamber only a few degrees above test temperature.

Optimum voltage settings for each test chamber were obtained with the aid of
a dual clock unit that can be plugged into the temperature controller. Each test
chamber has two available voltages. The lower voltage heats the test chamber to
slightly less than test temperature, while the high voltage heats the test chamber
slightly above test temperature. The voltages are adjusted with the aid of the
dual clock until the high voltage is on approximately 50 percent of the time.
The clock on the low voltage circuit runs continuously; the clock on the high
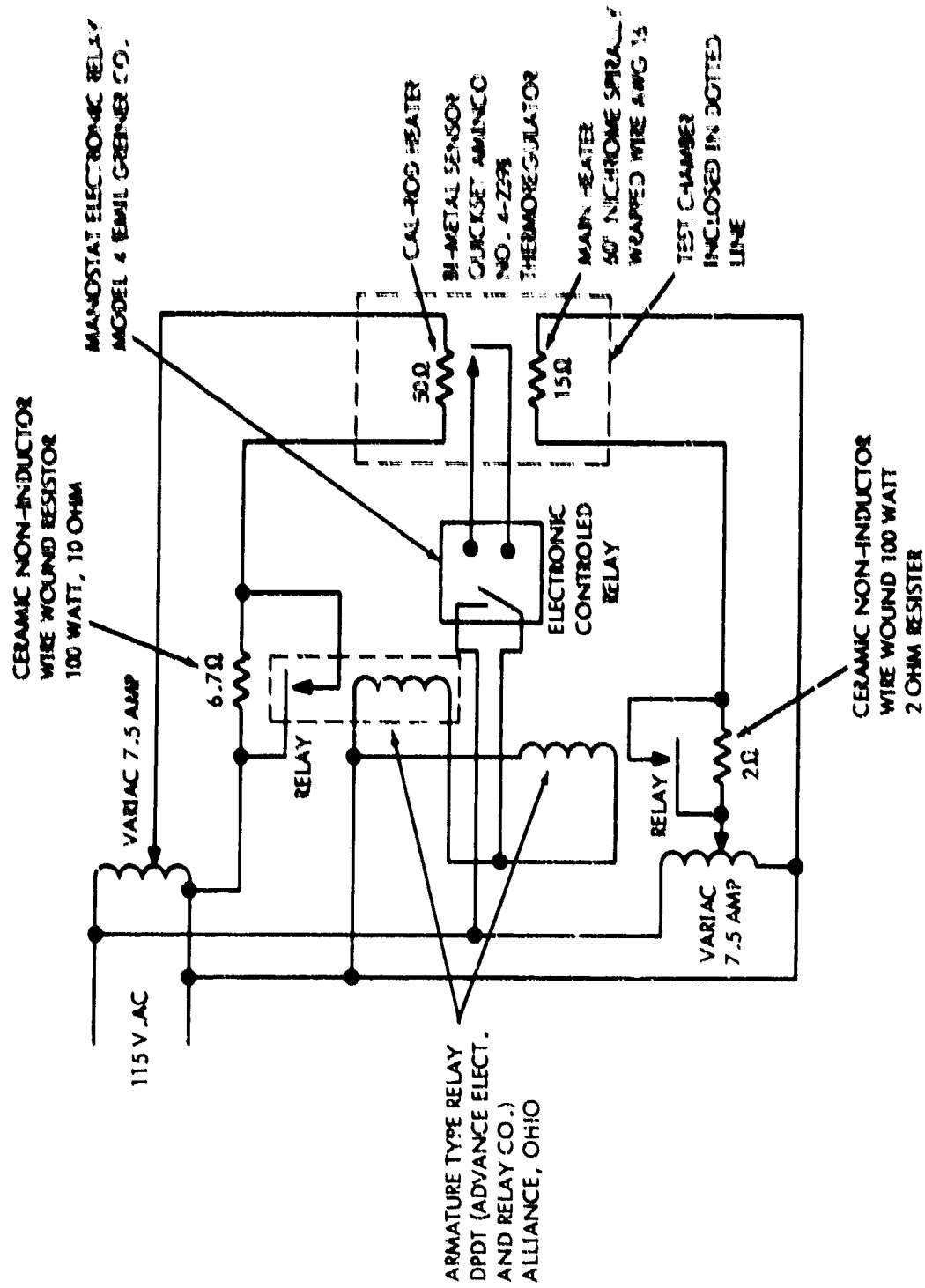voltage circuit operates only when this circuit is on.

FIG. 3 VACUUM STABILITY TEST CHAMBER TEMPERATURE CONTROL

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Figure 4 is a schematic of a new solid state electronic temperature controller that has recently been made and is being tested. It is believed that this new compact unit provides more precise temperature control than previous units.

TEMPERATURE MONITORING

Thermoelectric thermometry is now used to monitor all test chambers. Fiberglass duplex insulation #28 gage premium accuracy copper-constantan thermocouples (two in each test chamber) are connected to a selector switch box, a multipoint recorder, and a millivolt potentiometer. The two position switches of the selector switch box can be left in; the recorder position so that test temperatures can be continuously recorded, or can be switched to a millivolt potentiometer for accurate readings. Temperatures can be checked with extra thermocouples and portable potentiometers.

The two thermocouples are sealed into a glass unit similar to the sample glass unit. The bottom thermocouple is placed at the same depth as the sample, and the top thermocouple is mounted in the horizontal part of the capillary (about 2 inches out from sample tube) very nearly over the tubular heating element area. The bottom thermocouple has a length of rather tight spiral (springlike) just above the junction for a length of about three inches. The purpose of this spiral was to reduce heat radiation along the wire away from the junction.

The potentiometers have an external room temperature thermocouple junction with a well insulated calibrated thermometer. This system needs calibrating only several times a day.

DESCRIPTION OF GLASS TEST APPARATUS

Stability test units are fabricated from Pyrex glass by the Advanced Chemistry Division's glass blower. All tubes are annealed when made to relieve strains and to burn off organic matter on the surfaces of the glass. These sample test units are now made into a one-piece tube and manometer (Fig. 1).

1. Sample Test Unit

Straight walled 10 mm O.D. sample tubes of approximately 10 ml capacity are open at the top for sample insertion and sealed off flush with the horizontal top edge of the glass manometer. They are about 197 mm long after sealing. These sample tube test units are cut off from the manometers and discarded after each test whereas the manometers are used over again after cleaning.

The manometer part of the stability test unit is sealed onto the sample tube to complete the test unit. Manometers are made from $3/4$ to 1-$1/4$ mm I.D. bore capillary tubing. The opposite end of the manometer from the sample tube has a mercury well attached which is made from 20 mm O.D. glass tubing and is approximately 25 mm long. It is mounted at about a 52° angle. The vertical 920 mm length has a reference mark (from which pressure readings are taken) 820 mm up from the bottom. Horizontal part of the manometer one end of which is sealed to the sample tube is approximately 185 mm long with a calibration mark out 102 mm from the center of the vertical sample tube.
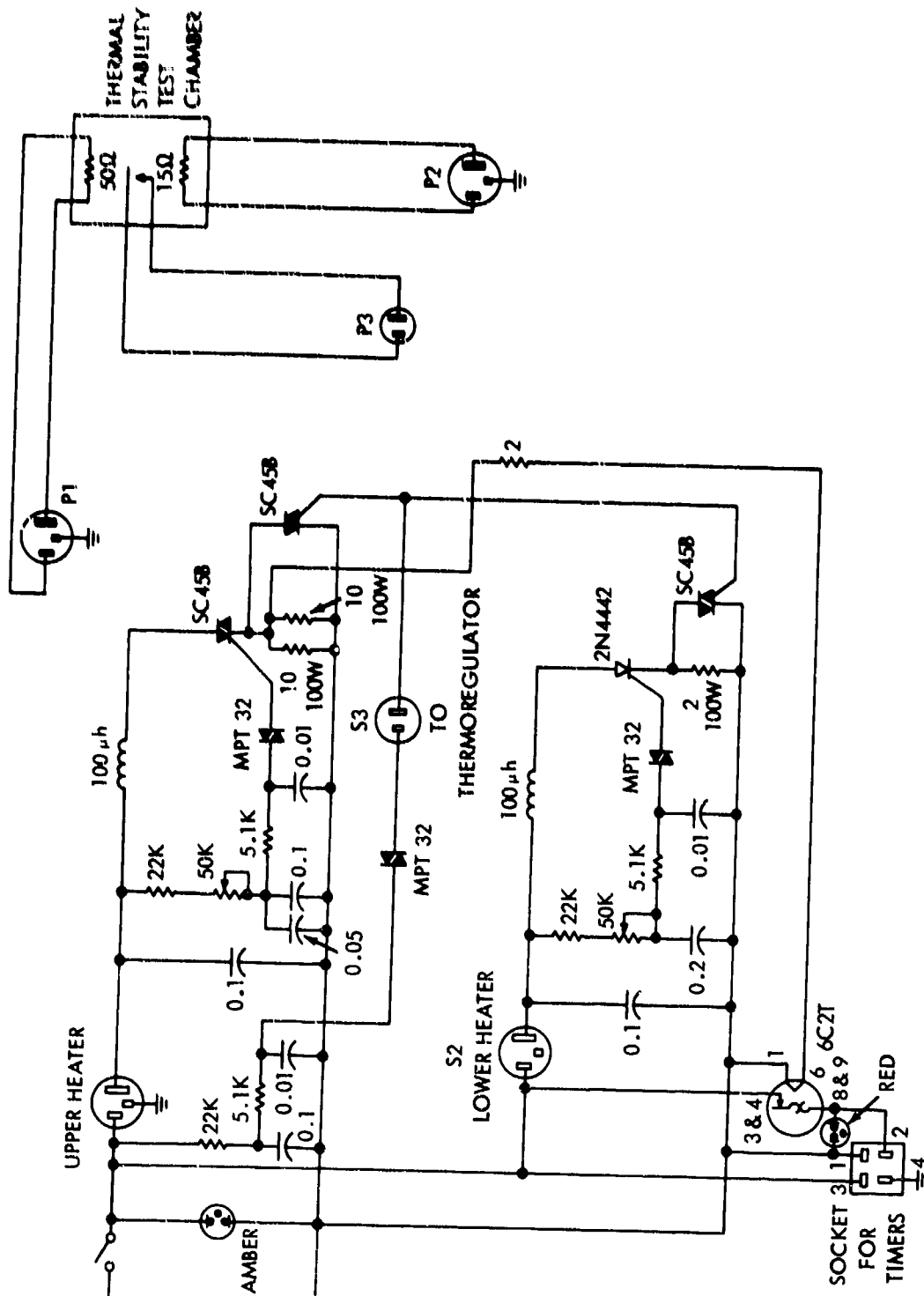
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FIG. 4 SCHEMATIC FOR SOLID STATE STABILITY TEST CHAMBER TEMPERATURE CONTROL

The hot or reactive zone of the sample test unit is that part completely in the test chamber during test. It consists of all the sample tube and that part of the horizontal section of the manometer out to the 102 mm mark. This is the only part of the test unit that is now calibrated. The cold zone of the manometer (that part from the 102 mm calibration mark to the vertical reference mark) is no longer calibrated since its volume is small compared to the 10 ml tube and is standard for each test.

2. Hot Zone Calibration

The volume of the hot zone of the test unit is calibrated by adding distilled water from a burette to the open end of the sample tube until the water level reaches the top of the tube (point where tube is sealed) and is allowed to flow up into the manometer to the 102 mm calibration mark. This volume minus the volume of the sample is the volume of the hot zone used in the calculation.

The sample units are drained of water after calibration and rinsed several times with acetone and thoroughly dried before each test. This drying is accomplished by hooking up the open end of the sample tube to a water aspirator and pulling dry air through the unit and at the same time heating the entire outer glass surfaces with a hand torch. A drying tube can be mounted to the mercury well to provide dry air during heating and cooling. Sample unit is now ready to be used for the test.

TEST METHODS AND PROCEDURES

This stability laboratory consists of three rooms, one large room and two isolated bays. The large room is used for sample preparation and weighing, and in each bay there are four test chambers in operation at all times. In one bay the low temperature stability and compatibility tests are run, while in the other bay all the high temperature stability tests are conducted.

Test procedures are essentially the same for stability and compatibility tests; however, since there are many different test temperatures in which time and temperature are the only variables the 260°C test used to screen most of the new heat resistant explosives will be described here.

Explosive compounds or compositions are usually crystalline, solid melts, or dry mechanical mixes and are easily weighed and transferred to the sample tube. Liquids and tacky samples are somewhat difficult to weigh and transfer so that different techniques are required as will be described later.

1. Setting Up Samples

Samples of 0.2 gram are weighed accurately on an analytical balance and transferred to the sample tube. The sample unit is then sealed with a torch and evacuated to 1 mm or less of mercury, and while the system is being pumped down 4 ml of mercury are added through the mercury well. Atmospheric pressure is allowed to enter the mercury well after the vacuum pump has been turned off, and the system now has a fluid mercury seal. Generally, test units are allowed to stand in the rack at room temperature overnight to test the system for leaks.

2. Conducting the Test

The test samples are placed in the 260°C test chamber after room temperature and barometric pressure readings are recorded. The distance in mm from the top of the mercury column to a reference mark is the zero time reading recorded. After 20 minutes of test readings are taken, then test continues for an additional 2-hour period. Pressure readings and barometric pressure are taken as often as necessary. A final reading is taken after 140 minutes of test (20-minute surge plus 2 hours). Samples are sometimes allowed to remain in the test chamber for comparison purposes as well as determining how long it takes each sample to decompose to the limit of the system. This additional information gained by leaving samples in longer than the 2-hour period is helpful in quality control work.

The first 20-minute period of test, referred to as the 20-minute surge, was at first discarded, and not calculated since the expansion of air in the system and other factors influence this initial surge and do not indicate true decomposition. Also, it takes 11 minutes for a 0.2 g sample to reach 260°C from room temperature.

The stability value for the 260°C test is based on the 2-hour period after the first 20 minutes of test. The volume of gas evolved from thermal decomposition during this period is calculated as ml of gas/gram of explosive/hour at 260°C by the simplified formula below.

3. Test Calculations

All calculations are corrected to standard temperature and pressure (0°C and 760 mm pressure). The volume of the sample is taken into consideration in the value of X in the formula.

$$V = \frac{(X)(Y)(Z)}{(W)(t)}$$

where:

V = ml of gas/gram of sample/hour at 260°C

X = volume of hot zone in ml

Y = conversion factor = $\dfrac{273}{533 \times 760}$

Z = corrected pressure (gas evolved in mm during 2-hour period)

W = sample weight in grams

t = heating time

4. Test Specifications

Heat resistant explosives that must stand aerodynamic heating during use were subjected to the 260°C stability test during their developmental stage. A limit of 2.0 ml gas/g/hr or less was set as an arbitrary stability value that they must not exceed. Tests have shown that this value was not set too high. Today these explosives have lower thermal stability limits written into their Naval Weapons Specifications. For example: Explosive HNS II WS 5003 has a maximum stability limit of 0.6 ml/g/hr for a 2-hour period. These lower limits were set so the Navy could be assured of high purity explosives.

Some of the latest heat resistant explosives discovered by the Advanced Chemistry Division of this laboratory are too stable at 260°C and must be tested at higher temperatures.

TEST EVALUATIONS

The first 20 minutes of test are influenced by many factors, some of which have been mentioned above. Surface moisture, and trapped occlusions can be considered dominant factors during this period.

Often samples are left in the test chamber after the 140-minute heating period where it can be determined how long it takes each sample to decompose to E.C. (exceeds capacity of the system). This outgassing between samples especially in quality control work is valuable in early development of compounds and in some cases may indicate impurities.

There are some explosive compounds that sublime during test, while others distill out vapors and liquids, also hot decomposition gases tend to escape to the cold zone of the system; however, the stability test chamber was designed to reduce this loss of sample during test. The manometer part of the hot zone is now heated 5 - 10° hotter than the sample, and sublimation stoppage in the cold zone of the manometer has just about been eliminated. Other techniques like those used on liquid explosives will be mentioned later in the report.

Vacuum Stability tests give results that are of a surveillance nature and are accurately a good indication of long term storage.

OTHER STABILITY TESTS

Standard military explosives, including underwater explosives used in mines and torpedoes, and some Navy plastic-bonded explosives (PBX) as well as most compatibility tests are generally conducted at 100°C. The test temperature is set by the environment of the explosive in a particular ordnance application. In the 100°C test a 2.0 ml gas/gram is the limit set for a 49-hour heating period. The first hour is discarded and the calculated stability value is for 48 hours.

Other PBX samples are tested at 150°C, one of which is NASA's ALSEP explosives developed at this laboratory while another is a liquid explosive.

Some explosives like those used in air to air missiles on fighter planes are tested at 200°C.

It is now possible to test liquid explosives by the vacuum stability test method and equipment at 150°C. In the past the Taliani type test was used only at lower temperatures. The test chamber design with its two heaters allows for heating the manometer part of the hot zone slightly hotter than the sample tube. This design reduces the gradient heat loss up the sample tube. In addition, a partial pressure of nitrogen makes it possible to test most liquids at higher temperatures than was possible in the past.

Special handling techniques were developed to handle viscous liquids and tacky samples. These materials are now weighed into a small calibrated vial (31 mm long, 7 mm O.D. and 4 mm I.D.) with a volume of approximately 0.65 ml. These vials containing the weighed sample are easily inserted into the sample tube and allowed to slide down to the bottom of the tube where the exact test temperature is maintained.

If an explosive passes the 100°C stability test it can be expected to be stable and pose no problem during use or storage. This test is directly related to the Navy surveillance tests.

COMPATIBILITY

Compatibility tests are necessary in designing a new piece of ordnance. These tests show which materials are compatible whether for use in a torpedo, mine, projectile, fuse, jet fighter plane or a guided missile. Concerned parties thoroughly discuss the problem involved and decide how best to design a compatibility test. Generally, the test is conducted under extreme conditions of temperature and weight ratio of explosive to sample. Samples that pass are considered safe to use and should offer no future problems.

Many new materials have been selected for use based on these tests. Possible inoperative ordnance devices and compatibility problems on long periods of storage are eliminated.

In this laboratory there is no standard compatibility test. The configuration of the material used in the proximity of the explosive in the piece of ordnance is the dominant factor in the physical shape of the sample to be tested. Each test is designed to simulate as near as possible the environment and conditions of the materials used in a particular ordnance application.

Preparation and handling of samples are often quite varied. Generally, solid materials are diced up into small squares of about 2 - 4 mm to increase surface area and increase contact with the explosive which would be somewhat more than would be the actual case. Tacky and viscous liquid samples are weighed into the small calibrated vials along with the explosives. These same vials can be used to separate the explosive from liquids and allow only vapor contact with the explosives during test. This type of compatibility test was one of the several types designed to evaluate sealants used on bomb and projectile fuses.

Most polymers and so-called cured plastics will give up sealed-in volatiles during test and it is essential in these cases to run the test under a partial pressure of nitrogen. This tends to hold down gases, volatiles, and liquids which when allowed to escape the reaction zone to the cold zone creates a situation where the system does not contain all the materials in the hot zone during the entire test. This partial pressure of nitrogen is a deterrent to this type of outgassing.

Compatibility tests on standard military explosives are conducted at 100°C. These procedures and equipment are identical to that used in the stability test. In the 100°C compatibility test the first hour is discarded and the compatibility value is based on 48 hours of heating at test temperature. Most tests are run as an explosive mixture (as compared to a composition explosive) and the calculated results are based on the weight of this mixture. Another method would be to base the calculation on the weight of the explosive only, and compare this with the known stability value for this explosive.

The prime importance of the compatibility test is evidence of chemical interaction exhibited by outgassing. If sample passes the test (2.0 ml/g/48 hours at 100°C or less), then no controls are necessary. Controls are only run on those samples which fail.

Most compatibility tests are evaluated with a weight ratio of 1/1 mixture of explosive and sample. These tests reveal borderline materials. Such compatibility tests were conducted by this laboratory for NASA's APOLLO space program. Here it was absolutely essential that all systems work.

Compatibility tests of two explosives or an explosive plus a solid are generally easily evaluated. These results can be compared directly with the stability value of the explosive. Other compatibility tests can be difficult. This would be the case in which an explosive is tested with a semisolid or a liquid. However, it should be pointed out that chemical interaction cannot always be detected in these compatibility tests. There is a need to know more about sample degradation during the compatibility test. Recently thin layer and vapor phase chromotography procedures have been used to measure residual explosive after the test (5). This laboratory plans to develop other methods to indicate chemical interaction during the compatibility test.

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13. ABSTRACT

This report covers a complete description of the methods and procedures used in the vacuum thermal stability test. It describes the construction and assembly of the test chamber, installation of the two heating elements, electronic temperature controller, thermocouple monitoring system, glass sample apparatus, sample tube calibration, and a compatibility test evaluation.

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